Unexpected Photoisomerization of a Pincer-type Amido Ligand Leads to Facial Coordination at Pt(IV).

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Supporting Information

General: All manipulations were carried out using standard Schlenk or glove box techniques under a dinitrogen atmosphere. Unless otherwise noted, solvents were deoxygenated and dried thoroughly by sparging with N_2 gas followed by passage through an activated alumina column. Non-halogenated solvents were typically tested with a standard purple solution of sodium benzophenone ketyl in tetrahydrofuran to confirm effective oxygen and moisture removal. The preparation of (BQA)PtMe was previously reported.[1] Other reagents were purchased from commercial vendors and used without further purification. Elemental analyses were performed by Desert Analytics, Tucson, Az. A Varian Mercury-300 NMR spectrometer or a Varian Inova-500 NMR spectrometer was used to record ^{1}H, ^{2}H, ^{13}C, and ^{19}F NMR spectra. 1D-NOE experiments were conducted using the Varian *GLIDE* software package. ^{1}H and ^{13}C NMR chemical shifts were referenced to residual solvent. ^{19}F NMR chemical shifts were referenced to an external C_6F_6 sample with a chemical shift of –165 ppm. Deuterated solvents were purchased from Cambridge Isotope Labs and were degassed and dried over activated 3 Å molecular sieves prior to use. Uv-vis spectra were collected on a Cary 50 spectrophotometer in CH_2Cl_2 with 1cm quartz cuvettes. IR measurements were obtained with a KBr solution cell using a Bio-Rad Excalibur FTS 3000 spectrometer controlled by Bio-Rad Merlin Software (v. 2.97) set at 4 cm^{-1} resolution. X-ray diffraction studies were carried out at the Beckman Institute Crystallographic Facility on a Brüker Smart 1000 CCD diffractometer and solved using SHELX v. 6.14.

Synthesis of (*mer*-BQA)Pt(CH$_3$)$_2$I / (*mer*-BQA)Pt(CH$_3$)(CD$_3$)I, 2: A solution of (BQA)PtMe (400 mg, 0.833 mmol), CH_3I (103 μL, 1.67 mmol), and acetone (50 mL) were combined in a glass reaction bomb, sealed with its Teflon stopcock, and heated to 70 °C for 18 h in the absence of light. The purple solution was evaporated to dryness under reduced pressure, and washed with petroleum ether (2 x 40 mL). Drying *in vacuo* of the powdery-purple solid (470 mg, 92%) yield a spectroscopically pure product. X-ray quality crystals were obtained by vapor diffusion of petroleum ether into acetone. Reactions with CD_3I proceeded under identical conditions. Characterization data for the all protio form: ^{1}H NMR (CD_2Cl_2, 300 MHz, 25 °C): δ 8.71 (m, $^2J_{PtH}$ = 41 Hz, 2H), 8.30 (d, 2H), 7.77 (d, 2H), 7.66 (t, 2H), 7.48 (m, 2H), 7.16 (d, 2H), 1.67 (s, $^2J_{PtH}$ = 60 Hz, 3H), 1.43 (s, $^2J_{PtH}$ = 70 Hz, 3H). ^{13}C NMR (CD_2Cl_2, 126 MHz, 25 °C): δ 155.1, 146.7, 140.3, 139.4, 133.4, 130.9, 122.3, 115.7, 115.6, 15.2 ($^1J_{PtC}$ = 550 Hz), 1.89 ($^1J_{PtC}$ = 582 Hz). UV-vis (nm (ε M^{-1} cm^{-1}), CH_2Cl_2): 292 (30000), 300 (27100), sh 396 (2900), 388 (3400), 534 (15500). IR (cm^{-1}, KBr): 3053 (vw), 2902 (w), 1582 (m), 1564 (s), 1496 (s), 1463 (s), 1400 (s), 1225 (w), 1180 (m), 1134 (m), 813 (m), 770 (m), 736 (m). Anal. Calcd. for $C_{20}H_{18}IN_3Pt$: C, 38.60; H, 2.92; N, 6.75. Found: C, 38.76; H, 3.15, N, 6.51.

Synthesis of (*fac*-BQA)Pt(CH$_3$)$_2$I / (*fac*-BQA)Pt(CH$_3$)(CD$_3$)I, 3: A solution of **2** (196 mg, 0.315 mmol) in acetone (20 mL) was added to a 200 mL reaction bomb, sealed with its Teflon stopcock, and placed 2" underneath a 100 watt incandescent light bulb for 48 h, with periodic agitation. During this period, the solution went from purple to red in color. The solvent was removed under reduced pressure affording a red solid which was washed with petroleum ether and dried *in vacuo* (196 mg, >99%). Quantitative conversion was observed by NMR spectroscopy. Reactions with (*mer*-BQA)Pt(CH_3)(CD_3)I proceeded under identical conditions. Characterization data for the all protio form: ^{1}H NMR (CD_2Cl_2, 300 MHz, 25 °C): δ 9.80 (m, $^3J_{PtH}$ =17 Hz, 2H), 8.25 (m, 2H), 7.96 (d, 2H), 7.58-7.47 (m, 4H), 7.40 (d, 2H), 1.25 (s {Pt-CH_3}, $^2J_{PtH}$ = 71 Hz, 6H). ^{13}C NMR (CD_2Cl_2, 126 MHz, 25 °C): δ 159.7, 150.2, 147.1, 138.3, 131.1, 129.1, 123.4, 122.7, 121.2, -8.0 ($^1J_{PtC}$ = 602 Hz). UV-vis (nm (ε M^{-1} cm^{-1}), CH_2Cl_2): 284 (15900), sh 298 (10600), sh 325 (4000), 422 (4700), sh 502 (1300). IR (cm^{-1}, KBr): 3051 (w), 2962 (w), 2897 (m), 1575 (m), 1565 (m), 1497 (s), 1458 (s), 1374 (s), 1308 (m), 1256 (m), 1224 (m), 1113 (m), 1056 (w), 1031 (w), 841 (w), 810 (m), 772 (m). Anal. Calcd. for $C_{20}H_{18}IN_3Pt$: C, 38.60; H, 2.92; N, 6.75. Found: C, 38.81; H, 3.18, N, 6.50.

Synthesis of [H(*fac*-BQA)Pt(CH$_3$)$_2$I][BF$_4$], 4. A 54 wt% solution of HBF_4 in ether (19 μL, 0.137 mmol) was added to a solution of **4** (81 mg, 0.130 mmol) in CH_2Cl_2 (7 mL) in a glass scintillation vial. The reaction was stirred for 2 h at 25 °C and the product was precipitated by addition of petroleum ether (13 mL). The rose colored solid (90 mg, 97%) was collected on a fritted glass funnel and dried *in vacuo*. The product was found to be pure by ^{1}H NMR spectroscopy and micro analysis. Crystals suitable for X-ray diffraction were obtained by diffusion of petroleum ether in to a concentrated solution of **4** in THF. ^{1}H NMR (CD_2Cl_2, 300 MHz, 25 °C): δ 10.33 (s {N-H}, $^2J_{PtH}$ = 21 Hz, 1H), 9.90 (m, $^3J_{PtH}$ =17 Hz, 2H), 8.78 (d, 2H), 8.50 (m, 2H), 7.96 (m, 2H), 7.83 (t, 2H), 7.74 (m, 2H) 1.54 (s {Pt-CH_3}, $^2J_{PtH}$ = 70 Hz, 6H). ^{13}C NMR (CD_2Cl_2, 126 MHz, 25 °C): δ 152.0, 144.8, 143.7, 139.9, 131.5, 130.5, 130.1, 129.9, 125.0, -12.41 ($^1J_{PtC}$ = 514 Hz). ^{19}F NMR (CD_2Cl_2, 282 MHz, 25°C): δ -147.4 (d, $^1J_{BF}$ = 15 Hz). UV-vis (nm (ε M^{-1} cm^{-1}), CH_2Cl_2): 302 (12900), 317 (10000), sh 240 (1700), 510 (630). IR (cm^{-1}, KBr): 3077 (br, N-H),

[1] Harkins, S. B.; Peters, J. C. *Organometallics* **2002**, *21*, 1753.

2978 (w), 2903 (w), 1510 (s), 1467 (m), 1398 (m), 1358 (m), 1221 (w), 1081 (s br, B-F), 835 (m), 798 (m), 769 (m) 734 (w). IR (cm^{-1}, CH_2Cl_2 sol. in KBr): 3127 (br, N-H), 2910 (w), 1591 (w), 1567 (w), 1513 1467 (w), 1359 (m), 1080 (s br), 1000 (m), 835 (m). Anal. Calcd. for $C_{20}H_{19}BF_4IN_3Pt$: C, 33.82; H, 2.70; N, 5.92. Found: C, 33.84; H, 2.46, N, 5.73.

Conversion of (BQA)Pt-Me to (BQA)Pt-Ph at rt: Under inert atmosphere an NMR tube was charged with **1** (20.0 mg, 0.042 mmol), $B(C_6F_5)_3$ (21.3 mg, 0.042 mmol), and C_6D_6 (~0.6 mL). ^1H NMR spectral analysis of the solution showed complete loss of the starting material methyl complex and quantitative production of previously characterized (BQA)Pt-Ph.[1]

Figure 1: Fully Labeled Drawing of (*mer*-BQA)$PtMe_2I$, **2** (hydrogen atoms omitted for clarity).

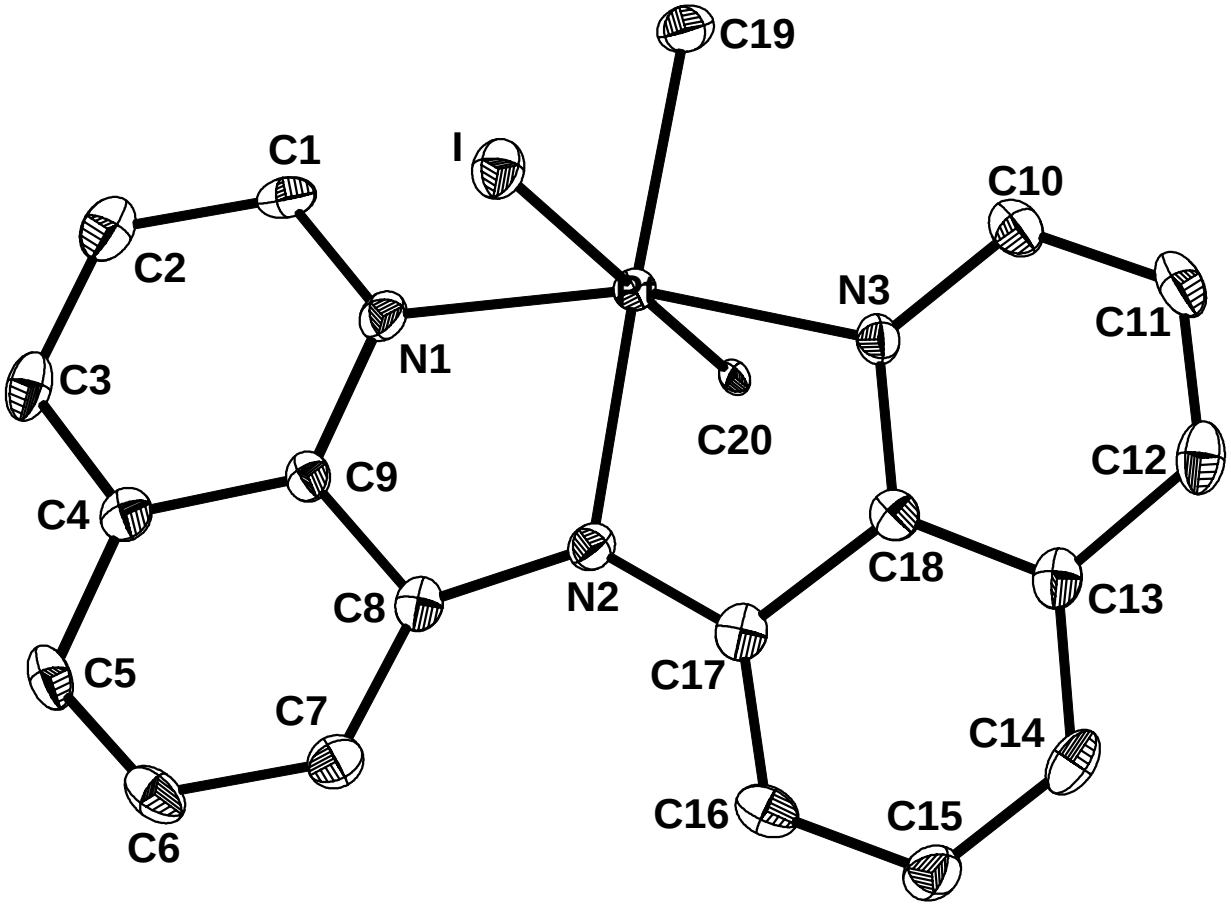

Figure 2: Fully Labeled Drawing of (*fac*-BQA)$PtMe_2I$, **3** (hydrogen atoms omitted for clarity).

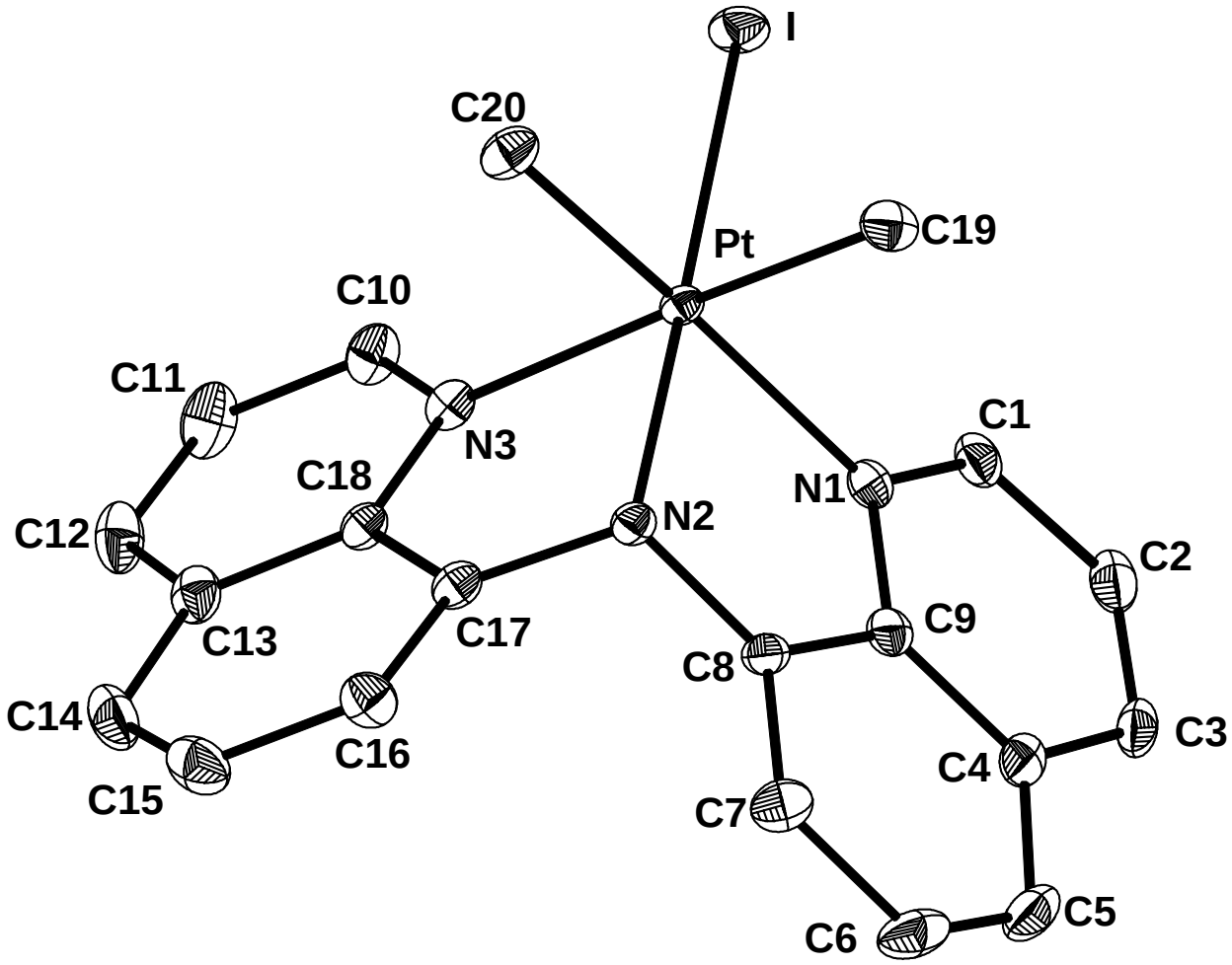

Figure 3: Fully Labeled Drawing of [H(*fac*-BQA)PtMe$_2$I][BF$_4$], **4** (hydrogen atoms attached to C omitted for clarity). Italicized atom labels correspond to atoms generated by symmetry transformation { x,-y+1/2,z }.

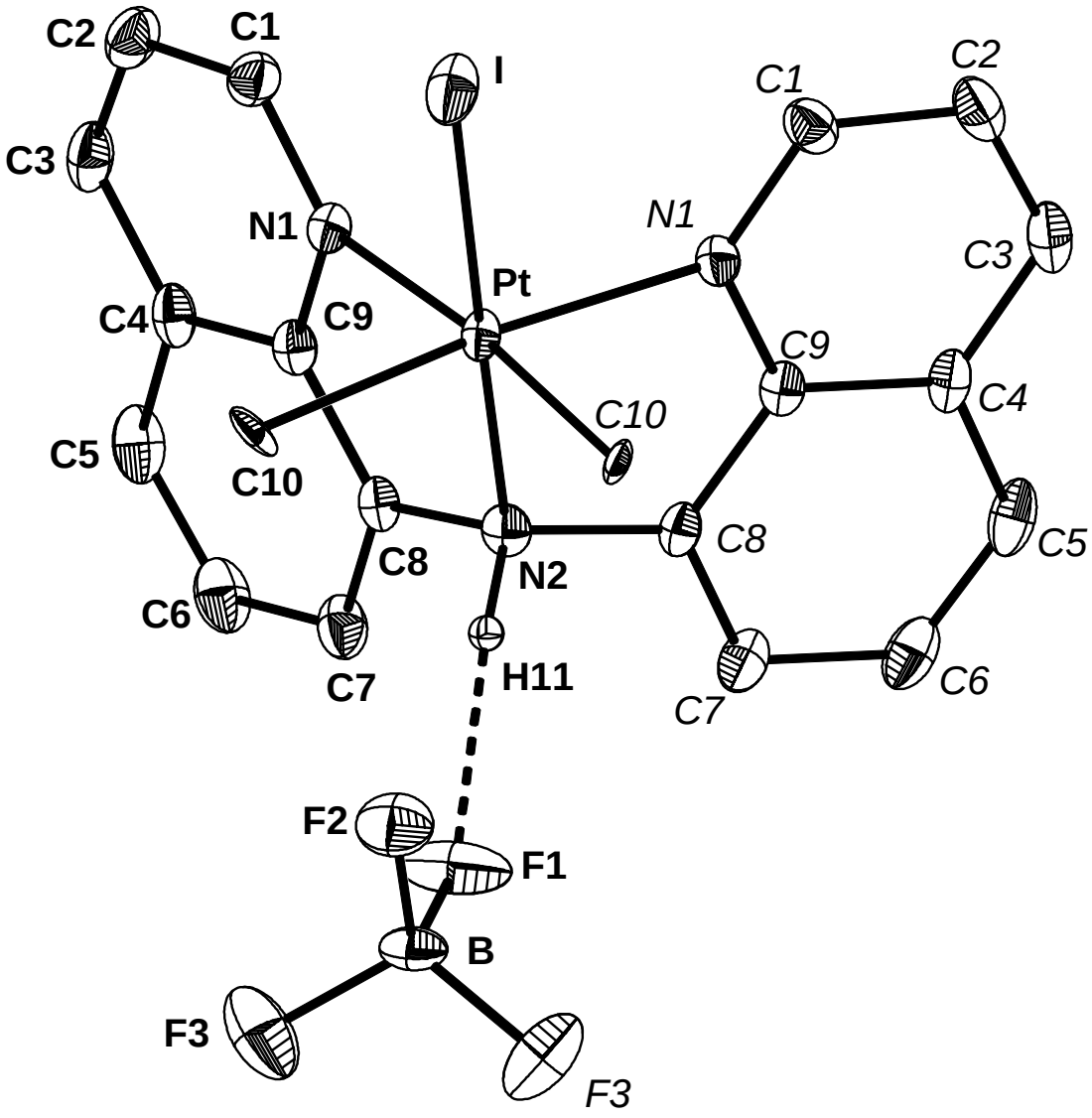

Table 1. Crystal data and structure refinement for **2**.

Name	(*mer*-BQA)$PtMe_2I$	
Empirical formula	$C_{20}H_{18}IN_3Pt$	
Formula weight	622.36	
Temperature	98(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/n (#14)	
Unit cell dimensions	a = 8.0242(10) Å	α= 90°
	b = 15.578(2) Å	β= 90.799(2)°
	c = 14.3744(18) Å	γ = 90°
Volume	1796.7(4) $Å^3$	
Z	4	
Density (calculated)	2.301 Mg/m^3	
Absorption coefficient	9.536 mm^{-1}	
F(000)	1160	
Crystal habit	block	
Crystal color	purple	
Crystal size	0.16 x 0.089 x 0.048 mm^3	
Theta range for data collection	1.93 to 28.46°	
Index ranges	-10<=h<=10, -20<=k<=19, -18<=l<=18	
Reflections collected	30699	
Independent reflections	4263 [R(int) = 0.0594]	
Completeness to theta = 28.46°	93.9 %	
Absorption correction	Semi-empirical from equivalents	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4263 / 0 / 229	
Goodness-of-fit on F^2	1.339	
Final R indices [I>2sigma(I)]	R1 = 0.0271, wR2 = 0.0604	
R indices (all data)	R1 = 0.0367, wR2 = 0.0633	
Extinction coefficient	0.00060(8)	
Largest diff. peak and hole	1.183 and -1.381 $e·Å^{-3}$	

Refinement Details

Refinement of F^2 against all reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2> 2sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on all data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

A slightly large prolate is evident in thermal ellipsoid of C20 which results from a partial population of I in this position arising from a trace quantity of the mirror image of **2** which has crystallized in an errant conformation. Refinement with a partial population of I in this position (< 3%) did not significantly improve the overall refinement and was not included as a part of the final refinement.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **2**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pt	1823(1)	1654(1)	2458(1)	11(1)
I	404(1)	3272(1)	2233(1)	18(1)
N(1)	1820(5)	1523(2)	1068(3)	15(1)
N(2)	-458(4)	1103(2)	2292(3)	13(1)
N(3)	1195(5)	1597(2)	3810(3)	14(1)
C(1)	3049(6)	1705(3)	483(3)	17(1)
C(2)	2858(6)	1627(3)	-482(3)	21(1)
C(3)	1358(6)	1357(3)	-839(3)	18(1)
C(4)	27(6)	1161(3)	-246(3)	15(1)
C(5)	-1560(6)	898(3)	-579(3)	17(1)
C(6)	-2768(6)	715(3)	47(3)	19(1)
C(7)	-2500(6)	762(3)	1008(3)	17(1)
C(8)	-960(5)	1011(3)	1383(3)	14(1)
C(9)	311(5)	1235(3)	727(3)	13(1)
C(10)	2047(6)	1881(3)	4547(3)	18(1)
C(11)	1466(6)	1769(3)	5458(3)	21(1)
C(12)	5(6)	1348(3)	5594(3)	20(1)
C(13)	-910(6)	1030(3)	4825(3)	16(1)
C(14)	-2413(6)	563(3)	4928(3)	18(1)
C(15)	-3193(6)	244(3)	4162(3)	20(1)
C(16)	-2601(5)	389(3)	3255(3)	17(1)
C(17)	-1192(5)	876(3)	3110(3)	14(1)
C(18)	-300(5)	1176(3)	3930(3)	13(1)
C(19)	4204(6)	2171(3)	2647(3)	17(1)
C(20)	2928(5)	438(3)	2607(3)	18(1)

Table 3. Bond lengths [Å] and angles [°] for **2**.

Pt-N(1)	2.008(4)	N(2)-Pt-C(19)	177.62(16)
Pt-N(3)	2.016(4)	N(1)-Pt-C(20)	90.22(16)
Pt-N(2)	2.033(4)	N(3)-Pt-C(20)	88.41(16)
Pt-C(19)	2.087(4)	N(2)-Pt-C(20)	90.53(15)
Pt-C(20)	2.102(5)	C(19)-Pt-C(20)	87.15(17)
Pt-I	2.7830(5)	N(1)-Pt-I	88.97(10)
N(1)-C(1)	1.336(6)	N(3)-Pt-I	92.59(10)
N(1)-C(9)	1.375(6)	N(2)-Pt-I	90.15(10)
N(2)-C(17)	1.368(6)	C(19)-Pt-I	92.18(13)
N(2)-C(8)	1.370(6)	C(20)-Pt-I	178.86(12)
N(3)-C(10)	1.329(6)	C(1)-N(1)-C(9)	119.9(4)
N(3)-C(18)	1.380(6)	C(1)-N(1)-Pt	127.9(3)
C(1)-C(2)	1.398(7)	C(9)-N(1)-Pt	112.2(3)
C(1)-H(1)	0.9500	C(17)-N(2)-C(8)	131.9(4)
C(2)-C(3)	1.369(7)	C(17)-N(2)-Pt	114.0(3)
C(2)-H(2)	0.9500	C(8)-N(2)-Pt	114.1(3)
C(3)-C(4)	1.409(7)	C(10)-N(3)-C(18)	119.8(4)
C(3)-H(3)	0.9500	C(10)-N(3)-Pt	128.4(3)
C(4)-C(5)	1.415(6)	C(18)-N(3)-Pt	111.8(3)
C(4)-C(9)	1.418(6)	N(1)-C(1)-C(2)	122.4(4)
C(5)-C(6)	1.361(7)	N(1)-C(1)-H(1)	118.8
C(5)-H(5)	0.9500	C(2)-C(1)-H(1)	118.8
C(6)-C(7)	1.397(6)	C(3)-C(2)-C(1)	118.8(5)
C(6)-H(6)	0.9500	C(3)-C(2)-H(2)	120.6
C(7)-C(8)	1.396(6)	C(1)-C(2)-H(2)	120.6
C(7)-H(7)	0.9500	C(2)-C(3)-C(4)	120.6(4)
C(8)-C(9)	1.442(6)	C(2)-C(3)-H(3)	119.7
C(10)-C(11)	1.406(7)	C(4)-C(3)-H(3)	119.7
C(10)-H(10)	0.9500	C(3)-C(4)-C(5)	123.0(4)
C(11)-C(12)	1.361(7)	C(3)-C(4)-C(9)	117.8(4)
C(11)-H(11)	0.9500	C(5)-C(4)-C(9)	119.2(4)
C(12)-C(13)	1.409(6)	C(6)-C(5)-C(4)	118.9(4)
C(12)-H(12)	0.9500	C(6)-C(5)-H(5)	120.6
C(13)-C(18)	1.400(6)	C(4)-C(5)-H(5)	120.6
C(13)-C(14)	1.417(6)	C(5)-C(6)-C(7)	122.8(4)
C(14)-C(15)	1.354(7)	C(5)-C(6)-H(6)	118.6
C(14)-H(14)	0.9500	C(7)-C(6)-H(6)	118.6
C(15)-C(16)	1.412(7)	C(8)-C(7)-C(6)	121.3(4)
C(15)-H(15)	0.9500	C(8)-C(7)-H(7)	119.4
C(16)-C(17)	1.380(6)	C(6)-C(7)-H(7)	119.4
C(16)-H(16)	0.9500	N(2)-C(8)-C(7)	130.1(4)
C(17)-C(18)	1.448(6)	N(2)-C(8)-C(9)	113.4(4)
C(19)-H(19A)	0.9800	C(7)-C(8)-C(9)	116.4(4)
C(19)-H(19B)	0.9800	N(1)-C(9)-C(4)	120.5(4)
C(19)-H(19C)	0.9800	N(1)-C(9)-C(8)	118.2(4)
C(20)-H(20A)	0.9800	C(4)-C(9)-C(8)	121.3(4)
C(20)-H(20B)	0.9800	N(3)-C(10)-C(11)	121.8(4)
C(20)-H(20C)	0.9800	N(3)-C(10)-H(10)	119.1
		C(11)-C(10)-H(10)	119.1
N(1)-Pt-N(3)	163.21(15)	C(12)-C(11)-C(10)	119.5(5)
N(1)-Pt-N(2)	81.48(15)	C(12)-C(11)-H(11)	120.3
N(3)-Pt-N(2)	81.80(15)	C(10)-C(11)-H(11)	120.3
N(1)-Pt-C(19)	99.05(17)	C(11)-C(12)-C(13)	119.8(5)
N(3)-Pt-C(19)	97.60(17)	C(11)-C(12)-H(12)	120.1

C(13)-C(12)-H(12)	120.1	N(3)-C(18)-C(13)	120.5(4)
C(18)-C(13)-C(12)	118.6(4)	N(3)-C(18)-C(17)	118.1(4)
C(18)-C(13)-C(14)	119.2(4)	C(13)-C(18)-C(17)	121.3(4)
C(12)-C(13)-C(14)	122.2(4)	Pt-C(19)-H(19A)	109.5
C(15)-C(14)-C(13)	119.2(4)	Pt-C(19)-H(19B)	109.5
C(15)-C(14)-H(14)	120.4	H(19A)-C(19)-H(19B)	109.5
C(13)-C(14)-H(14)	120.4	Pt-C(19)-H(19C)	109.5
C(14)-C(15)-C(16)	122.3(4)	H(19A)-C(19)-H(19C)	109.5
C(14)-C(15)-H(15)	118.8	H(19B)-C(19)-H(19C)	109.5
C(16)-C(15)-H(15)	118.8	Pt-C(20)-H(20A)	109.5
C(17)-C(16)-C(15)	121.0(4)	Pt-C(20)-H(20B)	109.5
C(17)-C(16)-H(16)	119.5	H(20A)-C(20)-H(20B)	109.5
C(15)-C(16)-H(16)	119.5	Pt-C(20)-H(20C)	109.5
N(2)-C(17)-C(16)	129.4(4)	H(20A)-C(20)-H(20C)	109.5
N(2)-C(17)-C(18)	113.7(4)	H(20B)-C(20)-H(20C)	109.5
C(16)-C(17)-C(18)	116.8(4)		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pt	12(1)	12(1)	11(1)	1(1)	0(1)	-1(1)
I	22(1)	15(1)	17(1)	1(1)	0(1)	2(1)
N(1)	19(2)	11(2)	14(2)	2(2)	0(2)	-1(2)
N(2)	15(2)	12(2)	13(2)	1(2)	1(2)	-1(2)
N(3)	17(2)	12(2)	12(2)	1(2)	-2(2)	0(2)
C(1)	16(2)	14(2)	21(2)	1(2)	1(2)	-5(2)
C(2)	26(3)	19(3)	19(2)	4(2)	3(2)	1(2)
C(3)	26(3)	15(2)	13(2)	3(2)	3(2)	7(2)
C(4)	20(2)	8(2)	17(2)	2(2)	-1(2)	2(2)
C(5)	24(2)	16(2)	12(2)	-3(2)	-5(2)	5(2)
C(6)	17(2)	16(2)	24(3)	-2(2)	-9(2)	3(2)
C(7)	16(2)	17(2)	18(2)	0(2)	2(2)	0(2)
C(8)	17(2)	10(2)	13(2)	-1(2)	1(2)	4(2)
C(9)	17(2)	9(2)	13(2)	1(2)	-3(2)	2(2)
C(10)	19(2)	18(2)	18(3)	-1(2)	-4(2)	1(2)
C(11)	26(3)	19(3)	16(2)	-4(2)	-8(2)	3(2)
C(12)	28(3)	19(2)	13(2)	0(2)	2(2)	8(2)
C(13)	21(2)	14(2)	14(2)	0(2)	1(2)	4(2)
C(14)	20(2)	20(3)	16(2)	4(2)	10(2)	4(2)
C(15)	14(2)	24(3)	22(3)	6(2)	0(2)	-1(2)
C(16)	13(2)	16(2)	23(3)	-4(2)	-2(2)	2(2)
C(17)	16(2)	11(2)	15(2)	-1(2)	2(2)	5(2)
C(18)	15(2)	8(2)	16(2)	1(2)	-3(2)	4(2)
C(19)	18(2)	15(2)	18(2)	0(2)	3(2)	-4(2)
C(20)	13(2)	31(3)	8(2)	7(2)	-5(2)	-14(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **2**.

	x	y	z	U(eq)
H(1)	4089	1893	730	20
H(2)	3754	1760	-882	25
H(3)	1212	1300	-1494	22
H(5)	-1779	848	-1228	21
H(6)	-3838	549	-179	23
H(7)	-3383	621	1414	20
H(10)	3076	2169	4457	22
H(11)	2090	1986	5973	25
H(12)	-398	1269	6207	24
H(14)	-2866	475	5527	22
H(15)	-4174	-89	4237	24
H(16)	-3182	148	2737	21
H(19A)	4678	1970	3240	26
H(19B)	4133	2799	2653	26
H(19C)	4918	1987	2137	26
H(20A)	2064	10	2727	26
H(20B)	3727	449	3129	26
H(20C)	3509	289	2034	26

Table 6. Crystal data and structure refinement for **3**.

Name	(*fac*-BQA)PtMe$_2$I	
Empirical formula	$C_{20}H_{18}IN_3Pt$	
Formula weight	622.36	
Temperature	98(2) K	
Wavelength	0.71073 Å	
Crystal habit	Rough block	
Crystal color	Red	
Crystal system	Monoclinic	
Space group	P 21/n (#14)	
Unit cell dimensions	a = 7.9108(8) Å	α= 90°
	b = 15.9623(16) Å	β= 90.771(2)°
	c = 14.5739(15) Å	γ = 90°
Volume	1840.1(3) Å^3	
Z	4	
Density (calculated)	2.246 Mg/m^3	
Absorption coefficient	9.310 mm^{-1}	
F(000)	1160	
Crystal size	0.15 x 0.15 x 0.11 mm^3	
Theta range for data collection	1.89 to 28.33°	
Index ranges	-10<=h<=10, -20<=k<=20, -19<=l<=19	
Reflections collected	26315	
Independent reflections	4305 [R(int) = 0.0386]	
Completeness to theta = 28.33°	93.8 %	
Absorption correction	Semi-empirical from equivalents	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4305 / 0 / 228	
Goodness-of-fit on F^2	1.087	
Final R indices [I>2sigma(I)]	R1 = 0.0202, wR2 = 0.0466	
R indices (all data)	R1 = 0.0234, wR2 = 0.0474	
Largest diff. peak and hole	1.331 and -0.830 e·Å^{-3}	

Refinement Details

Refinement of F^2 against all reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2> 2sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on all data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 7. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **3**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pt	1425(1)	3738(1)	7439(1)	10(1)
I	28(1)	4497(1)	8839(1)	17(1)
N(1)	1225(3)	4737(2)	6468(2)	12(1)
N(2)	2416(3)	3148(2)	6330(2)	12(1)
N(3)	-756(3)	3044(2)	6990(2)	13(1)
C(1)	578(4)	5490(2)	6562(2)	16(1)
C(2)	770(5)	6121(2)	5900(3)	20(1)
C(3)	1698(4)	5957(2)	5139(2)	19(1)
C(4)	2390(4)	5155(2)	5006(2)	17(1)
C(5)	3291(4)	4915(2)	4218(2)	22(1)
C(6)	3902(4)	4121(3)	4142(2)	23(1)
C(7)	3666(4)	3521(2)	4838(2)	18(1)
C(8)	2780(4)	3717(2)	5620(2)	13(1)
C(9)	2138(4)	4553(2)	5700(2)	14(1)
C(10)	-2341(4)	3066(2)	7272(2)	18(1)
C(11)	-3572(4)	2497(2)	6976(3)	23(1)
C(12)	-3124(4)	1866(2)	6397(3)	22(1)
C(13)	-1436(4)	1810(2)	6082(2)	17(1)
C(14)	-863(5)	1181(2)	5474(3)	22(1)
C(15)	778(5)	1185(2)	5204(3)	22(1)
C(16)	1915(4)	1813(2)	5493(2)	17(1)
C(17)	1406(4)	2453(2)	6060(2)	13(1)
C(18)	-291(4)	2425(2)	6390(2)	13(1)
C(19)	3709(4)	4265(2)	7791(2)	17(1)
C(20)	1930(4)	2733(2)	8270(2)	17(1)

Table 8. Bond lengths [Å] and angles [°] for **3**.

Pt-N(2)	2.036(3)	C(19)-Pt-N(1)	85.19(12)
Pt-C(20)	2.046(3)	N(2)-Pt-N(3)	80.54(10)
Pt-C(19)	2.052(3)	C(20)-Pt-N(3)	85.74(12)
Pt-N(1)	2.137(3)	C(19)-Pt-N(3)	171.87(12)
Pt-N(3)	2.146(3)	N(1)-Pt-N(3)	97.51(10)
Pt-I	2.6299(3)	N(2)-Pt-I	177.72(7)
N(1)-C(1)	1.315(4)	C(20)-Pt-I	89.02(10)
N(1)-C(9)	1.372(4)	C(19)-Pt-I	89.70(10)
N(2)-C(8)	1.409(4)	N(1)-Pt-I	98.16(7)
N(2)-C(17)	1.421(4)	N(3)-Pt-I	97.47(7)
N(3)-C(10)	1.325(4)	C(1)-N(1)-C(9)	119.4(3)
N(3)-C(18)	1.373(4)	C(1)-N(1)-Pt	129.7(2)
C(1)-C(2)	1.403(5)	C(9)-N(1)-Pt	110.2(2)
C(1)-H(1)	0.9500	C(8)-N(2)-C(17)	114.8(3)
C(2)-C(3)	1.364(5)	C(8)-N(2)-Pt	111.7(2)
C(2)-H(2)	0.9500	C(17)-N(2)-Pt	111.11(19)
C(3)-C(4)	1.406(5)	C(10)-N(3)-C(18)	118.7(3)
C(3)-H(3)	0.9500	C(10)-N(3)-Pt	130.6(2)
C(4)-C(9)	1.411(5)	C(18)-N(3)-Pt	110.2(2)
C(4)-C(5)	1.414(5)	N(1)-C(1)-C(2)	122.6(3)
C(5)-C(6)	1.361(6)	N(1)-C(1)-H(1)	118.7
C(5)-H(5)	0.9500	C(2)-C(1)-H(1)	118.7
C(6)-C(7)	1.409(5)	C(3)-C(2)-C(1)	119.0(3)
C(6)-H(6)	0.9500	C(3)-C(2)-H(2)	120.5
C(7)-C(8)	1.382(5)	C(1)-C(2)-H(2)	120.5
C(7)-H(7)	0.9500	C(2)-C(3)-C(4)	120.1(3)
C(8)-C(9)	1.433(5)	C(2)-C(3)-H(3)	119.9
C(10)-C(11)	1.396(5)	C(4)-C(3)-H(3)	119.9
C(10)-H(10)	0.9500	C(3)-C(4)-C(9)	117.5(3)
C(11)-C(12)	1.363(5)	C(3)-C(4)-C(5)	124.1(3)
C(11)-H(11)	0.9500	C(9)-C(4)-C(5)	118.4(3)
C(12)-C(13)	1.420(5)	C(6)-C(5)-C(4)	120.1(3)
C(12)-H(12)	0.9500	C(6)-C(5)-H(5)	119.9
C(13)-C(18)	1.404(5)	C(4)-C(5)-H(5)	119.9
C(13)-C(14)	1.419(5)	C(5)-C(6)-C(7)	121.6(3)
C(14)-C(15)	1.361(5)	C(5)-C(6)-H(6)	119.2
C(14)-H(14)	0.9500	C(7)-C(6)-H(6)	119.2
C(15)-C(16)	1.408(5)	C(8)-C(7)-C(6)	120.9(3)
C(15)-H(15)	0.9500	C(8)-C(7)-H(7)	119.6
C(16)-C(17)	1.377(4)	C(6)-C(7)-H(7)	119.6
C(16)-H(16)	0.9500	C(7)-C(8)-N(2)	124.8(3)
C(17)-C(18)	1.433(4)	C(7)-C(8)-C(9)	117.5(3)
C(19)-H(19A)	0.9800	N(2)-C(8)-C(9)	117.6(3)
C(19)-H(19B)	0.9800	N(1)-C(9)-C(4)	121.3(3)
C(19)-H(19C)	0.9800	N(1)-C(9)-C(8)	117.2(3)
C(20)-H(20A)	0.9800	C(4)-C(9)-C(8)	121.5(3)
C(20)-H(20B)	0.9800	N(3)-C(10)-C(11)	123.0(3)
C(20)-H(20C)	0.9800	N(3)-C(10)-H(10)	118.5
		C(11)-C(10)-H(10)	118.5
N(2)-Pt-C(20)	91.93(12)	C(12)-C(11)-C(10)	119.1(3)
N(2)-Pt-C(19)	92.36(12)	C(12)-C(11)-H(11)	120.4
C(20)-Pt-C(19)	90.58(14)	C(10)-C(11)-H(11)	120.4
N(2)-Pt-N(1)	81.06(10)	C(11)-C(12)-C(13)	120.0(3)
C(20)-Pt-N(1)	171.63(12)	C(11)-C(12)-H(12)	120.0

C(13)-C(12)-H(12)	120.0
C(18)-C(13)-C(14)	118.9(3)
C(18)-C(13)-C(12)	117.3(3)
C(14)-C(13)-C(12)	123.8(3)
C(15)-C(14)-C(13)	119.5(3)
C(15)-C(14)-H(14)	120.3
C(13)-C(14)-H(14)	120.3
C(14)-C(15)-C(16)	121.6(3)
C(14)-C(15)-H(15)	119.2
C(16)-C(15)-H(15)	119.2
C(17)-C(16)-C(15)	121.1(3)
C(17)-C(16)-H(16)	119.4
C(15)-C(16)-H(16)	119.4
C(16)-C(17)-N(2)	125.3(3)
C(16)-C(17)-C(18)	117.5(3)
N(2)-C(17)-C(18)	117.2(3)
N(3)-C(18)-C(13)	121.9(3)
N(3)-C(18)-C(17)	116.9(3)
C(13)-C(18)-C(17)	121.2(3)
Pt-C(19)-H(19A)	109.5
Pt-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
Pt-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
Pt-C(20)-H(20A)	109.5
Pt-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	109.5
Pt-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5

Table 9. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pt	10(1)	11(1)	9(1)	0(1)	2(1)	1(1)
I	20(1)	19(1)	13(1)	-3(1)	5(1)	3(1)
N(1)	10(1)	13(1)	14(1)	-2(1)	-2(1)	0(1)
N(2)	10(1)	14(1)	11(1)	0(1)	2(1)	0(1)
N(3)	12(1)	13(1)	14(1)	2(1)	2(1)	2(1)
C(1)	16(2)	15(2)	18(2)	-2(1)	-4(1)	-3(1)
C(2)	21(2)	13(2)	24(2)	2(1)	-8(1)	-3(1)
C(3)	20(2)	16(2)	20(2)	7(1)	-8(1)	-9(1)
C(4)	12(2)	21(2)	17(2)	4(1)	-5(1)	-7(1)
C(5)	19(2)	29(2)	18(2)	9(2)	0(1)	-8(2)
C(6)	17(2)	38(2)	14(2)	4(2)	5(1)	-2(2)
C(7)	15(2)	23(2)	16(2)	0(1)	5(1)	3(1)
C(8)	12(2)	18(2)	10(2)	1(1)	0(1)	-3(1)
C(9)	11(2)	17(2)	14(2)	-1(1)	-1(1)	-4(1)
C(10)	17(2)	16(2)	22(2)	2(1)	5(1)	1(1)
C(11)	12(2)	22(2)	34(2)	4(2)	5(1)	0(1)
C(12)	15(2)	18(2)	31(2)	3(2)	-3(1)	-4(1)
C(13)	18(2)	12(2)	20(2)	3(1)	-2(1)	1(1)
C(14)	26(2)	13(2)	26(2)	-3(1)	-5(2)	-1(1)
C(15)	28(2)	17(2)	21(2)	-6(1)	-1(2)	5(1)
C(16)	16(2)	17(2)	18(2)	-3(1)	2(1)	2(1)
C(17)	13(2)	13(2)	13(2)	3(1)	1(1)	2(1)
C(18)	13(2)	12(2)	13(2)	3(1)	0(1)	1(1)
C(19)	12(2)	24(2)	15(2)	-2(1)	0(1)	-2(1)
C(20)	19(2)	17(2)	16(2)	4(1)	2(1)	4(1)

Table 10. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **3**.

	x	y	z	U(eq)
H(1)	-45	5612	7098	20
H(2)	261	6654	5982	23
H(3)	1878	6385	4698	23
H(5)	3469	5308	3739	26
H(6)	4502	3968	3607	27
H(7)	4122	2974	4769	22
H(10)	-2654	3489	7696	22
H(11)	-4706	2549	7174	27
H(12)	-3940	1464	6205	26
H(14)	-1617	760	5257	26
H(15)	1165	753	4810	27
H(16)	3053	1795	5294	21
H(19A)	4508	4181	7291	26
H(19B)	4150	4000	8351	26
H(19C)	3558	4867	7898	26
H(20A)	2446	2285	7909	26
H(20B)	875	2529	8536	26
H(20C)	2711	2903	8764	26

Table 11. Crystal data and structure refinement for **4**.

Name	[H(*fac*-BQA)PtMe$_2$I][BF$_4$]	
Empirical formula	[$C_{20}H_{19}IN_3Pt$][BF_4]	
Formula weight	710.18	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal habit	rough block	
Crystal color	red-orange	
Crystal system	Orthorhombic	
Space group	P n m a (#62)	
Unit cell dimensions	a = 13.8342(5) Å	α= 90°
	b = 14.8844(5) Å	β= 90°
	c = 9.9174(4) Å	γ = 90°
Volume	2042.13(13) Å^3	
Z	4	
Density (calculated)	2.310 Mg/m^3	
Absorption coefficient	8.431 mm^{-1}	
F(000)	1328	
Crystal size	0.20 x 0.15 x 0.10 mm^3	
Theta range for data collection	2.47 to 42.79°	
Index ranges	-22<=h<=25, -28<=k<=28, -17<=l<=15	
Reflections collected	40673	
Independent reflections	7049 [R(int) = 0.0618]	
Completeness to theta = 42.79°	92.0 %	
Absorption correction	Semi-empirical from equivalents	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7049 / 0 / 147	
Goodness-of-fit on F^2	1.032	
Final R indices [I>2sigma(I)]	R1 = 0.0497, wR2 = 0.1253	
R indices (all data)	R1 = 0.0816, wR2 = 0.1453	
Largest diff. peak and hole	7.090 and -6.237 e·Å^{-3}	

Refinement Details

Refinement of F^2 against all reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F^2> 2sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on all data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

A slightly large prolate is evident in thermal ellipsoid of C10 which results from what is presumed to be a partial population of I due to a trace impurity in this position. Refinement with a partial population of I in this position (< 4%) did not significantly improve the overall refinement and was not included as a part of the final refinement. Hydrogen bonding between F1 and N2 is also evident and summarized in Table 16.

Table 12. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **4**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pt	5709(1)	2500	1981(1)	13(1)
N(1)	4656(3)	3563(2)	1902(4)	16(1)
N(2)	5257(3)	2500	4040(6)	15(1)
C(10)	6825(3)	3496(3)	2203(5)	22(1)
I	6194(1)	2500	-549(1)	25(1)
C(1)	4353(3)	4023(3)	845(5)	20(1)
C(2)	3795(3)	4813(3)	971(6)	23(1)
C(3)	3552(3)	5122(3)	2242(6)	22(1)
C(4)	3848(3)	4620(3)	3387(5)	18(1)
C(5)	3627(4)	4888(3)	4729(6)	24(1)
C(6)	3938(4)	4382(3)	5796(6)	26(1)
C(7)	4493(4)	3597(3)	5585(5)	22(1)
C(8)	4712(3)	3324(2)	4298(4)	15(1)
C(9)	4400(3)	3833(3)	3182(4)	14(1)
B	7704(6)	2500	6080(8)	23(1)
F(1)	6693(4)	2500	6059(6)	45(2)
F(2)	8046(4)	2500	4737(5)	30(1)
F(3)	8027(3)	3270(3)	6714(5)	43(1)

Table 13. Bond lengths [Å] and angles [°] for **4**.

Pt-N(2)	2.135(5)	C(1)-N(1)-C(9)	119.9(4)
Pt-N(1)#1	2.151(4)	C(1)-N(1)-Pt	128.8(3)
Pt-N(1)	2.151(4)	C(9)-N(1)-Pt	110.8(3)
Pt-C(10)#1	2.151(4)	C(8)#1-N(2)-C(8)	114.0(4)
Pt-C(10)	2.151(4)	C(8)#1-N(2)-Pt	108.6(3)
Pt-I	2.5978(5)	C(8)-N(2)-Pt	108.6(3)
N(1)-C(1)	1.320(6)	C(8)#1-N(2)-F(1)	103.8(3)
N(1)-C(9)	1.378(6)	C(8)-N(2)-F(1)	103.8(3)
N(2)-C(8)#1	1.462(5)	Pt-N(2)-F(1)	118.2(2)
N(2)-C(8)	1.462(5)	C(8)#1-N(2)-H(11)	108.5
N(2)-F(1)	2.821(7)	C(8)-N(2)-H(11)	108.5
N(2)-H(11)	0.9300	Pt-N(2)-H(11)	108.5
C(10)-H(10A)	0.9800	F(1)-N(2)-H(11)	9.7
C(10)-H(10B)	0.9800	Pt-C(10)-H(10A)	109.5
C(10)-H(10C)	0.9800	Pt-C(10)-H(10B)	109.5
C(1)-C(2)	1.412(7)	Pt-C(10)-H(10C)	109.5
C(1)-H(1)	0.9500	N(1)-C(1)-C(2)	122.4(5)
C(2)-C(3)	1.383(8)	N(1)-C(1)-H(1)	118.8
C(2)-H(2)	0.9500	C(2)-C(1)-H(1)	118.8
C(3)-C(4)	1.420(7)	C(3)-C(2)-C(1)	119.3(5)
C(3)-H(3)	0.9500	C(3)-C(2)-H(2)	120.3
C(4)-C(9)	1.414(6)	C(1)-C(2)-H(2)	120.3
C(4)-C(5)	1.423(7)	C(2)-C(3)-C(4)	118.9(4)
C(5)-C(6)	1.368(8)	C(2)-C(3)-H(3)	120.5
C(5)-H(5)	0.9500	C(4)-C(3)-H(3)	120.5
C(6)-C(7)	1.414(7)	C(9)-C(4)-C(3)	118.5(4)
C(6)-H(6)	0.9500	C(9)-C(4)-C(5)	118.9(4)
C(7)-C(8)	1.373(6)	C(3)-C(4)-C(5)	122.6(4)
C(7)-H(7)	0.9500	C(6)-C(5)-C(4)	120.1(4)
C(8)-C(9)	1.409(6)	C(6)-C(5)-H(5)	119.9
B-F(3)	1.381(6)	C(4)-C(5)-H(5)	119.9
B-F(3)#1	1.381(6)	C(5)-C(6)-C(7)	120.7(5)
B-F(1)	1.398(10)	C(5)-C(6)-H(6)	119.6
B-F(2)	1.414(9)	C(7)-C(6)-H(6)	119.6
		C(8)-C(7)-C(6)	120.1(5)
N(2)-Pt-N(1)#1	80.62(13)	C(8)-C(7)-H(7)	119.9
N(2)-Pt-N(1)	80.62(13)	C(6)-C(7)-H(7)	119.9
N(1)#1-Pt-N(1)	94.67(19)	C(7)-C(8)-C(9)	120.2(4)
N(2)-Pt-C(10)#1	96.46(17)	C(7)-C(8)-N(2)	121.7(4)
N(1)#1-Pt-C(10)#1	89.03(14)	C(9)-C(8)-N(2)	118.1(4)
N(1)-Pt-C(10)#1	174.84(15)	N(1)-C(9)-C(8)	119.2(3)
N(2)-Pt-C(10)	96.46(17)	N(1)-C(9)-C(4)	120.9(4)
N(1)#1-Pt-C(10)	174.84(15)	C(8)-C(9)-C(4)	119.9(4)
N(1)-Pt-C(10)	89.03(14)	F(3)-B-F(3)#1	112.1(7)
C(10)#1-Pt-C(10)	87.1(2)	F(3)-B-F(1)	109.3(4)
N(2)-Pt-I	177.94(13)	F(3)#1-B-F(1)	109.3(4)
N(1)#1-Pt-I	98.00(10)	F(3)-B-F(2)	108.7(4)
N(1)-Pt-I	98.01(10)	F(3)#1-B-F(2)	108.7(4)
C(10)#1-Pt-I	85.02(15)	F(1)-B-F(2)	108.7(6)
C(10)-Pt-I	85.02(15)	B-F(1)-N(2)	135.6(5)

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+1/2,z

Table 14. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **4**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pt	15(1)	10(1)	15(1)	0	3(1)	0
N(1)	18(2)	12(1)	17(2)	1(1)	-1(1)	0(1)
N(2)	8(2)	17(2)	20(2)	0	0(1)	0
C(10)	24(2)	9(1)	34(2)	-13(1)	-22(2)	9(1)
I	38(1)	17(1)	21(1)	0	11(1)	0
C(1)	17(2)	19(2)	24(2)	4(1)	-4(1)	-1(1)
C(2)	20(2)	18(2)	31(2)	6(2)	-4(2)	3(1)
C(3)	17(2)	13(1)	36(3)	2(2)	1(2)	1(1)
C(4)	14(2)	12(1)	27(2)	-2(1)	3(1)	0(1)
C(5)	21(2)	16(2)	34(3)	-6(2)	11(2)	2(1)
C(6)	29(2)	21(2)	27(3)	-8(2)	10(2)	-2(2)
C(7)	27(2)	16(2)	22(2)	-3(1)	5(2)	-2(1)
C(8)	15(2)	11(1)	19(2)	-2(1)	1(1)	-1(1)
C(9)	11(1)	13(1)	20(2)	-1(1)	2(1)	-1(1)
B	25(3)	31(3)	11(3)	0	-2(2)	0
F(1)	24(2)	89(5)	21(2)	0	-5(2)	0
F(2)	31(2)	40(3)	20(2)	0	5(2)	0
F(3)	45(2)	39(2)	46(2)	-22(2)	8(2)	-9(2)

Table 15. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for **4**.

	x	y	z	U(eq)
H(11)	5804	2500	4584	18
H(10A)	6550	4095	2061	33
H(10B)	7334	3387	1536	33
H(10C)	7099	3459	3112	33
H(1)	4516	3815	-31	24
H(2)	3588	5129	190	28
H(3)	3194	5662	2347	26
H(5)	3263	5419	4884	29
H(6)	3780	4561	6688	31
H(7)	4714	3258	6335	26

Table 16. Hydrogen bonds for **4** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(2)-H(11)...F(1)	0.93	1.91	2.821(7)	165.6

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+1/2,z